Report
Laboratory Pre-Program Performance Evaluation Sample Study - Sediment
Newark Bay Study Area
Risk Assessment Sampling

Prepared by

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Prepared for

TIERRA SOLUTIONS, INC.

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LIST OF ACRONYMS AND ABBREVIATIONS

EDS	Environmental Data Services, Ltd.
ERA	Environmental Resource Associates
GPC	gel permeation chromatography
IRMM	Institute for Reference Materials and Measurements
mg/kg	milligrams per kilogram
NA	not applicable
NBSA	Newark Bay Study Area
NELAC	National Environmental Laboratory Accreditation Conference
ng/g	nanograms per gram
ng/kg	nanograms per kilogram
ng/ml	nanograms per milliliter
NIST	National Institute of Standards and Technology
PAHs	polycyclic aromatic hydrocarbons
PCBs	polychlorinated biphenyls
PCDDs/PCDFs	polychlorinated dibenzo-*p*-dioxins/polychlorinated dibenzofurans
PE	performance evaluation
QAPP	Quality Assurance Project Plan
SIM	selective ion monitoring
SOP	Standard Operating Procedure
TAL	target analyte list
μg/kg	micrograms per kilogram
USEPA	United States Environmental Protection Agency

1. INTRODUCTION

A laboratory pre-program performance evaluation (PE) sample study was developed and implemented in support of the Newark Bay Study Area (NBSA) risk assessment sampling. The analytical procedures included in this pre-program PE study were selected based upon their inclusion in the in-program PE programs implemented by the Cooperating Parties Group during the Lower Passaic River Restoration Project (Windward 2009a, 2009b). A summary of the analytical groups included in the NBSA risk assessment sampling sediment pre-program PE study is provided in Table 1-1 below, along with a comparison of the analytical groups included in the Lower Passaic River Restoration Project.

Table 1-1
Analytical Groups Included
in the Sediment Pre-program PE Study

Analytical Groups	Included in NBSA Sediment Risk Assessment Sampling	Included in Lower Passaic River Restoration Project
PCDDs/PCDFs	x	x
PCB Congeners	x	x
Aroclor PCBs		
TAL Metals	x	x
Mercury	x	x
Methylmercury	x	x
Hexavalent Chromium		
Semivolatile Organics SIM	x	x[1]
Semivolatile Organics		x[1]
Volatile Organics		
Percent Moisture		
Pesticides	x	x
Chlorinated Herbicides		
Butyltins	x[2]	
Total Petroleum Hydrocarbons		
Total Organic Carbon		
pH		
Oxidation Reduction Potential		
Total Sulfide		
Total Phosphorus		
Total Kjeldahl Nitrogen		
Acid Volatile Sulfide/Simultaneously Extracted Metals		
Ammonia		
Cyanide		
Grain Size		

[1] The Lower Passaic River Restoration Project PE program only evaluated a subset of semivolatile organics, PAHs, via two methodologies, semivolatile organics SIM and semivolatile high resolution mass spectrometry (the latter represented by semivolatile organics in Table 1-1). For the NBSA risk assessment sampling, PAHs were only reported via semivolatile organics SIM.
[2] Butyltins were added to the initial scope and will be reported separately.

The scope of the PE study was submitted to the USEPA in a document titled Newark Bay Study Area Clam/Crab, Fish and Sediment Quality Triad Quality Assurance Project Plans (QAPPs) Pre-Program Performance Evaluation Sample Study dated June 24, 2014 (Attachment A; Tierra 2014a). The scope of the study was approved by the USEPA prior to implementation.

The pre-program PE sample study described in Tierra 2014a provides for the evaluation of the analytical procedures in both sediment and tissue matrices. However, this report is limited to the sediment PE sample component of the overall study.

A PE sample is a sample of known analyte concentration(s) submitted “blind” to the laboratory, i.e., the composition is unknown to the laboratory or analyst. The “blind” PE sample is identified, handled, and submitted for analysis in a manner consistent with field samples collected for analysis per the specifications in the associated QAPP, thereby limiting, to the extent possible, the laboratory’s ability to distinguish the PE sample from field samples submitted for analysis. Further, a “blind” sample is one for which source and certified analyte concentrations of the PE sample are not shared with the laboratory prior to reporting of sample results and therefore supports an assessment of the laboratory’s ability to produce results within the acceptance limits associated with that PE sample. Pre-program PE studies are intended to thoroughly verify laboratory performance with sufficient time to address any problems that may be identified before initiating field sample collection. This pre-program PE study was performed in advance of sample collection activities in order to establish laboratory proficiency in the use of project specific analytical procedures applied to the sediment/solid sample type. Any unsatisfactory results reported by a given laboratory were used to identify the source of the deficiency and to institute appropriate corrective actions. Sediment PE samples were submitted to, and analyzed by, the laboratories in June 2014 through September 2014.

2. SEDIMENT PRE-PROGRAM PERFORMANCE EVALUATION STUDY PROCEDURES

Sediment PE samples were obtained from various venders as identified in Table 2-1 below. The PE sample is either a natural sediment/soil matrix containing chemical constituents at known concentrations or the natural sediment/soil matrix fortified to provide specific analytical profiles.

All sediment PE samples prepared for use in this study are categorized as blind. To simulate as accurately as possible the submittal of a “real” sample to the laboratory, all necessary sample container labels, identification, and chain of custody were completed by EDS (an entity external to the laboratory), consistent with the specifications for the field program, and sent directly to the laboratory for analysis. Table 2-1, below, contains details regarding the sediment pre-program PE sample distribution to participating laboratories.

Table 2-1
Sediment Matrix
Pre-Program Performance Evaluation Sample Summary

Analytical Group	Analytical Method	Vendor Name / Catalog Number	Laboratory
PCDDs/PCDFs	USEPA Method 1613B	NIST/1944	eurofins/Lancaster Laboratories
PCB Congeners	USEPA Method 1668A	NIST/1944	eurofins/Lancaster Laboratories
Organochlorine Pesticides	USEPA Method 1699	Phenova/Lot # 7051-14	Vista Analytical Laboratories
Semivolatile Organics (SIM)	USEPA Method Modified SW-846 8270D	NIST/1944	eurofins/Lancaster Laboratories
Mercury	USEPA Method 1631	NRC/MESS-3	eurofins/Frontier Global Sciences
Methylmercury	USEPA Method 1630	IRMM/CC-580	eurofins/Frontier Global Sciences
TAL Metals	USEPA Method SW-846 6020	ERA/540	eurofins/Lancaster Laboratories

Upon receipt of PE sample results from the laboratories, EDS compared those results to the acceptance limits associated with each PE sample. Adequate laboratory proficiency is demonstrated when reported results are within the established acceptance limits. Alternatively, results that do not meet established acceptance limits are critically reviewed to identify appropriate corrective actions necessary to render the analytical system acceptable for analysis of field samples. The results reported, acceptance limits, and follow-up corrective actions for each of the sediment pre-program PE samples submitted for analysis are located in Section 3 of this report.

3. SEDIMENT PRE-PROGRAM PE SAMPLE RESULTS AND DISCUSSION

This section of the report provides detailed sediment PE sample results obtained by each of the participating laboratories for each sediment PE sample as outlined in the June 24, 2014 document to the USEPA.

3.1 PCDDs/PCDFs

Performance evaluation results for PCDDs/PCDFs in the sediment matrix are summarized in Table 3.1-1 below. All results reported by eurofins/Lancaster Laboratories, Lancaster, PA were within the established acceptance limits.

Table 3.1-1
PCDDs/PCDFs - Sediment
Analytical Method/SOP Reference: L-1[a]

Analyte	Certified Value ng/kg	Acceptance Limits[b] ng/kg	Reported Concentration ng/kg	Pass / Fail
2378-TCDD	133	100 - 166	133	Pass
12378-PeCDD	19.0	14 - 24	19.2	Pass
123478-HxCDD	26.0	20 - 33	24.5	Pass
123678-HxCDD	56.0	42 - 70	59.0	Pass
123789-HxCDD	53.0	40 - 66	41.9	Pass
1234678-HpCDD	800	600 - 1000	860	Pass
OCDD	5800	4350 - 7250	5680	Pass
2378-TCDF	39.0	29 - 49	34.5	Pass
12378-PeCDF	45.0	34 - 56	45.7	Pass
23478-PeCDF	45.0	34 - 56	48.5	Pass
123478-HxCDF	220	165 - 275	202	Pass
123678-HxCDF	90.0	68 - 113	91.4	Pass
234678-HxCDF	54.0	41 - 68	49.6	Pass
1234678-HpCDF	1000	750 - 1250	1000	Pass
1234789-HpCDF	40.0	30 - 50	43.5	Pass
OCDF	1000	750 - 1250	1080	Pass

[a] As defined in Worksheet #23-1 of the Crab and Clam Sampling and Analysis QAPP Rev. 3a, August 2014 (Tierra 2014b)
[b] The acceptance limits are defined on Worksheet #12-2 of the Crab and Clam Sampling and Analysis QAPP Rev. 3a, August 2014.

3.2 PCB Congeners

Performance evaluation results for PCB congeners in the sediment matrix are summarized in Table 3.2-1 below. Results reported by eurofins/Lancaster Laboratories, Lancaster, PA for all analytes were within the established acceptance limits with five exceptions. PCB-66, PCB-95, PCB-170, PCB-194 and PCB-206 had reported values that were less than the lower acceptance limits during the initial analysis of the PE sample.

Table 3.2-1
PCB Congeners - Sediment
Analytical Method/SOP Reference: L-2[a]

Analyte	Certified Value µg/kg	Acceptance Limits[b] µg/kg	First PE Sample Reported Concentration µg/kg	Pass / Fail	Second PE Sample Reported Concentration µg/kg	Pass / Fail
PCB-8	22300	16725 - 27875	18900	Pass	21300	Pass
PCB-31	78700	59025 - 98375	81000	Pass	73500	Pass
PCB-52	79400	59550 - 99250	80300	Pass	65400	Pass
PCB-66	71900	53925 - 89875	47600	Fail	70400	Pass
PCB-95	65000	48750 - 81250	45900	Fail	46800	Fail
PCB-99	37500	28125 - 46875	29300	Pass	32100	Pass
PCB-105	24500	18375 - 30625	22100	Pass	21800	Pass
PCB-118	58000	43500 - 72500	49100	Pass	56000	Pass
PCB-170	22600	16950 - 28250	14000	Fail	18300	Pass
PCB-183	12190	9143 - 15238	10400	Pass	Co-eluted with PCB-185	NA
PCB-187	25100	18825 - 31375	25100	Pass	28300	Pass
PCB-194	11200	8400 - 14000	7520	Fail	10100	Pass
PCB-195	3750	2813 - 4688	3260	Pass	3820	Pass
PCB-206	9210	6908 - 11513	6730	Fail	9360	Pass
PCB-209	6810	5108 - 8513	5840	Pass	7300	Pass

[a] As defined in Worksheet #23-1 of the Crab and Clam Sampling and Analysis QAPP Rev. 3a, August 2014
[b] The acceptance limits are defined on Worksheet #12-2 of the Crab and Clam Sampling and Analysis QAPP Rev. 3a, August 2014.

A corrective action review was initiated to investigate the cause of these failures. Two procedural improvements were recommended as a result of this investigation: the addition of initial calibration standards at multiple concentration levels across the linear range of the instrument for all target analytes, and the incorporation of fourteen additional carbon-labeled analog standards to both the calibration standards and the samples prior to extraction.

It was determined that performance would be improved if the laboratory performed the initial calibration at six concentration levels for all 209 PCB congeners versus the former initial calibration where only the twelve dioxin-like PCBs and the earliest and latest eluted congener at each level of chlorination were calibrated using standards at six concentration levels, with the remaining congeners calibrated using only a single concentration level. The laboratory implemented the six level calibration procedure for all 209 congeners as part of its corrective action and the new calibration standards can be found in Attachment B. A comparison of the former calibration procedure and that implemented as part of this corrective action for the five failed PCB congeners is provided in Table 3.2-2 below.

Table 3.2-2
Comparison of Calibration Levels
for the Five Failed PCB Congeners
Prior to and After Corrective Action

	Calibration Levels Prior to Corrective Action Review ng/ml						**Calibration Levels After Corrective Action Review ng/ml**					
Analyte	**CSL**	**CS1**	**CS2**	**CS3**	**CS4**	**CS5**	**CSL**	**CS1**	**CS2**	**CS3**	**CS4**	**CS5**
PCB-66	---	---	---	50	---	---	1.0	4.0	10	50	200	600
PCB-95	---	---	---	50	---	---	1.0	4.0	10	50	200	600
PCB-170	---	---	---	50	---	---	1.0	4.0	10	50	200	600
PCB-194	---	---	---	75	---	---	1.5	6.0	15	75	300	900
PCB-206	0.20	1.0	5.0	50	400	2000	1.5	6.0	15	75	300	900

CSL - extended calibration low level solution
CS1 - calibration standard one
CS2 - calibration standard two
CS3 - calibration standard three
CS4 - calibration standard four
CS5 - calibration standard five

It was also determined that the inclusion of more carbon-labeled analog standards would improve the laboratory's ability to accurately identify and quantitate target PCB congeners in complex matrices. Table 3.2-3 below lists the additional carbon-labeled analog standards that were added to all standards and samples prior to extraction as the second part of the corrective action. The additional carbon-labeled analog standards were identified by EDS, for use by the laboratory, based upon the large body of experience EDS has had with PCB congener analysis in sediment sample matrices, and the noted improvement of overall data quality when these additional carbon-labeled analog standards are included.

Table 3.2-3
PCB Congeners
Additional Carbon-Labeled Analog Standards

13C12-PCB-8	13C12-PCB-60	13C12-PCB-127	13C12-PCB-162
13C12-PCB-31	13C12-PCB-70	13C12-PCB-128	13C12-PCB-180
13C12-PCB-32	13C12-PCB-85	13C12-PCB-133	
13C12-PCB-47	13C12-PCB-95	13C12-PCB-141	

A second PE sample was then submitted to the laboratory after implementing the corrective actions described above. Upon evaluation of the results, analyte PCB-95 continued to fail with results below the lower acceptance limit. Also, PCB-183 and PCB-185 were reported as a co-elution, although they had been resolved and reported separately during the first PE sample analysis. The laboratory was then provided with the certificate of analysis for the material used as the PE sample and asked to determine possible further corrective action.

The review of PCB-95 results was not able to identify any non-conformance or other analytical failure. All quality control data associated with this analyte were within acceptance criteria.

The laboratory indicated after further review of chromatography from each of two instruments that PCBs 183 and 185 cannot be consistently resolved on a routine basis. Therefore these two PCBs will be reported as a co-eluting pair (PCB 183/185) for the NBSA risk assessment samples analyzed.

3.3 Organochlorine Pesticides

Performance evaluation results for organochlorine pesticides in the sediment matrix are summarized in Table 3.3-1 below. All results reported by Vista Analytical Laboratory, El Dorado Hills, CA, were within the established acceptance limits for each analyte.

Table 3.3-1
Organochlorine Pesticides - Sediment
Analytical Method/SOP Reference: L-11[a]

Analyte	Certified Value µg/kg	Acceptance Limits[b] µg/kg	Reported Concentration µg/kg	Pass / Fail
alpha-BHC	446000	127000 - 558000	358000	Pass
Lindane (gamma-BHC)	347000	88600 - 427000	259000	Pass
beta-BHC	221000	39200 - 314000	229000	Pass
delta-BHC	149000	27900 - 206000	120000	Pass
Heptachlor	62900	15700 - 87100	46400	Pass
Aldrin	342000	96900 - 425000	312000	Pass
cis-Heptachlor Epoxide	233000	73100 - 284000	163000	Pass
trans-Chlordane (gamma)	124000	40700 - 154000	90700	Pass
cis-Chlordane (alpha)	234000	72900 - 290000	177000	Pass
Endosulfan I (alpha)	320000	48800 - 352000	134000	Pass
4,4'-DDE	454000	155000 - 585000	401000	Pass
Dieldrin	205000	59300 - 256000	157000	Pass
Endrin	157000	36200 - 398000	93300	Pass
Endosulfan II (beta)	394000	72200 - 434000	165000	Pass
4,4'-DDD	339000	115000 - 452000	311000	Pass
4,4'-DDT	366000	89900 - 508000	338000	Pass
Endosulfan Sulfate	445000	87000 - 561000	265000	Pass
4,4'-Methoxychlor	247000	24700 - 412000	253000	Pass
Endrin Aldehyde	362000	36200 - 398000	259000	Pass
Endrin Ketone	314000	67700 - 375000	227000	Pass

[a] As defined in Worksheet #23-1 of the Crab and Clam Sampling and Analysis QAPP Rev. 3a, August 2014
[b] The PT Performance Acceptance Limits are calculated using the regression equations and fixed acceptance criteria specified in the USEPA National Standards Criteria Document and/or the NELAC proficiency testing requirements.

3.4 Semivolatile Organics SIM

Performance evaluation results for semivolatile organics SIM in the sediment matrix are summarized in Table 3.4-1 below. All results reported by eurofins/Lancaster Laboratories, Lancaster, PA from the first PE sample analysis were outside the established acceptance limits for each analyte with the exceptions of benzo(k)fluoranthene and chrysene. All failed results were below the lower acceptance limits except for dibenzo(a,h)anthracene, which exceeded the higher acceptance limit. A corrective action review was initiated to investigate the cause of these failures.

Table 3.4-1
Semivolatile Organics SIM - Sediment
Analytical Method/SOP Reference: L-9, L-30, L-31[a]

Analyte	Certified Value µg/kg	Acceptance Limits[b] µg/kg	First PE Sample Reported Concentration µg/kg	Pass / Fail	Second PE Sample Reported Concentration µg/kg	Pass / Fail
Benzo(a)anthracene	4720	3540 - 5900	2900	Fail	4000	Pass
Benzo(a)pyrene	4300	3225 - 5375	2400	Fail	3100	Fail
Benzo(b)fluoranthene	3870	2903 - 4838	2600	Fail	4500	Pass
Benzo(e)pyrene	3280	2460 - 4100	2300	Fail	3100	Pass
Benzo(g,h,i)perylene	2840	2130 - 3550	1900	Fail	2300	Pass
Benzo(k)fluoranthene	2300	1725 - 2875	2700	Pass	3200	Fail
Chrysene	4860	3645 - 6075	3900	Pass	5700	Pass
Dibenzo(a,h)anthracene	424	318 - 530	540	Fail	700	Fail
Fluoranthene	8920	6690 - 11150	5900	Fail	8400	Pass
Indeno(1,2,3-cd)pyrene	2780	2085 - 3475	2000	Fail	2700	Pass
Perylene	1170	878 - 1463	630	Fail	780	Fail
Phenanthrene	5270	3953 - 6588	3700	Fail	5000	Pass
Pyrene	9700	7275 - 12125	5600	Fail	8200	Pass

[a] As defined in Worksheet #23-1 of the Crab and Clam Sampling and Analysis QAPP Rev. 3a, August 2014
[b] The acceptance limits on Worksheet #12-2 of the Crab and Clam Sampling and Analysis QAPP Rev. 3a, August 2014 are defined as recovery within limits set by the PE sample vendor. However, acceptance limits of 75-125% of certified values were used in this evaluation as these limits are consistent with acceptance limits used for this same PE material when assessing performance of PCDDs/PCDFs and PCB congeners, and acceptance limits were not provided by the vendor for this material.

It was determined that a gel permeation chromatography (GPC) cleanup was not performed on the extract for this analysis. The laboratory agreed that the extracts for all future sediment samples submitted for analysis would undergo this process.

A second PE sample was then submitted to the laboratory following implementation of the corrective action. Upon evaluation, all results were within the established limits for each analyte with four exceptions: benzo(a)pyrene, benzo(k)fluoranthene, dibenzo(a,h)anthracene, and perylene. Values for benzo(a)pyrene and perylene were both lower than the lower acceptance limits while results for benzo(k)fluoranthene and dibenzo(a,h)anthracene were higher than the higher acceptance limits. The laboratory was then provided with the certificate of analysis for the material used as the PE sample and asked to determine possible further corrective action.

This second review concluded that benzo(k)fluoranthene and dibenzo(a,h)anthracene were higher than the acceptance limits due to co-elution with benzo(j)fluoranthene and dibenzo(a,c)anthracene, respectively. Both benzo(j)fluoranthene and dibenzo(a,c)anthracene were identified by the manufacturer as present in the material used as the PE sample. To address this issue, the laboratory will report all results for benzo(k)fluoranthene and dibenzo(a,h)anthracene with a comment that states the values represent the total of both the target analyte and the co-eluting isomer.

The review of data for perylene and benzo(a)pyrene for the second PE sample did not identify any non-conformance or other analytical failure. All quality control data associated with these analytes were within acceptance criteria. Although results reported do not meet the established acceptance limits, no additional corrective actions were identified.

3.5 Mercury

Performance evaluation results for mercury in the sediment matrix are summarized in Table 3.5-1 below. The mercury result reported by eurofins/Frontier Global Sciences, Bothell, WA, was within the established acceptance limits for the analyte.

Table 3.5-1
Mercury - Sediment
Analytical Method/SOP Reference: L-4[a]

Analyte	Certified Value ng/g	Acceptance Limits[b] ng/g	Reported Concentration ng/g	Pass / Fail
Mercury	91	68 - 114	88	Pass

[a] As defined in Worksheet #23-1 of the Crab and Clam Sampling and Analysis QAPP Rev. 3a, August 2014
[b] The acceptance limits are defined on Worksheet #12-2 of the Crab and Clam Sampling and Analysis QAPP Rev. 3a, August 2014.

3.6 Methylmercury

Performance evaluation results for methylmercury in the sediment matrix are summarized in Table 3.6-1 below. The methylmercury result reported by eurofins/Frontier Global Sciences, Bothell, WA, was within the established acceptance limits for the analyte.

Table 3.6-1
Methylmercury - Sediment
Analytical Method/SOP Reference: L-5[a]

Analyte	Certified Value ng/kg	Acceptance Limits[b] ng/kg	Reported Concentration ng/kg	Pass / Fail
Methylmercury	75	49 - 101	68	Pass

[a] As defined in Worksheet #23-1 of the Crab and Clam Sampling and Analysis QAPP Rev. 3a, August 2014
[b] The acceptance limits are defined on Worksheet #12-2 of the Crab and Clam Sampling and Analysis QAPP Rev. 3a, August 2014.

3.7 TAL Metals

Performance evaluation results for TAL metals in the sediment matrix are summarized in Table 3.7-1 below. All results reported by eurofins/Lancaster Laboratories, Lancaster, PA, were within the established acceptance limits for each analyte.

Table 3.7-1
TAL Metals - Sediment
Analytical Method/SOP Reference: L-3[a]

Analyte	Certified Value mg/kg	Acceptance Limits[b] mg/kg	Reported Concentration mg/kg	Pass / Fail
Aluminum	9390	4080 - 14700	11000	Pass
Antimony	129	28.9 - 322	81.8	Pass
Arsenic	88.4	61.0 - 116	106	Pass
Barium	210	154 - 266	228	Pass
Beryllium	55.8	40.8 - 70.9	65.4	Pass
Cadmium	143	104 - 182	155	Pass
Calcium	7530	5620 - 9440	8550	Pass
Chromium	86.8	60.0 - 114	90.7	Pass
Cobalt	199	148 - 250	232	Pass
Copper	268	204 - 332	306	Pass
Iron	12800	4040 - 21500	13900	Pass
Lead	97.9	69.3 - 126	103	Pass
Magnesium	2850	1860 - 3840	3280	Pass
Manganese	425	324 - 525	480	Pass
Nickel	236	175 - 302	266	Pass
Potassium	2570	1570 - 3570	2990	Pass
Selenium	127	84.6 - 170	142	Pass
Silver	66.2	44.4 - 88.0	84.2	Pass
Sodium	1040	628 - 1450	1100	Pass
Thallium	140	95.6 - 185	150	Pass
Vanadium	157	112 - 202	174	Pass
Zinc	130	87.0 - 173	155	Pass

[a] As defined in Worksheet #23-1 of the Crab and Clam Sampling and Analysis QAPP Rev. 3a, August 2014

[b] The PT Performance Acceptance Limits are calculated using the regression equations and fixed acceptance criteria specified in the USEPA National Standards Criteria Document and/or the NELAC proficiency testing requirements.

4. CONCLUSIONS

Sediment matrix pre-program PE samples were evaluated for seven analytical groups. The pre-program PE study for the sediment matrix demonstrated that the laboratories can successfully identify and quantitate a representative list of analytes planned for the NBSA risk assessment sampling, with the exception of PCB-95, benzo(a)pyrene and perylene. Two semivolatile organics will be reported as co-elutions, benzo(k)fluoranthene with benzo(j)fluoranthene and dibenzo(a,h)anthracene with dibenzo(a,c)anthracene, rather than individual results.

5. REFERENCES

Tierra. 2014a. Newark Bay Study Area. Clam/Crab, Fish and Sediment Quality Triad Quality Assurance Project Plans, Pre-Program Performance Evaluation Sample Study. June 24.

Tierra. 2014b. Newark Bay Study Area. Quality Assurance Project Plan. Crab and Clam Sampling and Analysis. Final. Revision 3a, August 2014.

Windward. 2009a. Lower Passaic River Restoration Project. Quality Assurance Project Plan. Fish and Decapod Crustacean Tissue Collection for Chemical Analysis and Fish Community Survey. Final. August 6.

Windward. 2009b. Lower Passaic River Restoration Project. Quality Assurance Project Plan. Surface Sediment Chemical Analyses and Benthic Invertebrate Toxicity and Bioaccumulation and Tissue Testing. Final. October 8.

Attachment A

Newark Bay Study Area
Clam/Crab, Fish and Sediment Quality Triad[1] Quality Assurance Project Plans
Pre-Program Performance Evaluation Sample Study
(Prepared by Environmental Data Services/Tierra Solutions Inc.; June 24, 2014)

1. Introduction

A Laboratory Pre-Program Performance Evaluation (PE) study has been developed for the Newark Bay Study Area (NBSA) Clam/Crab, Fish, and Sediment Quality Triad[1] Quality Assurance Project Plans (QAPPs). Tierra Solutions Inc. (Tierra) will implement the study during the summer 2014, prior to project start. Details of the laboratory pre-program study are provided below.

2. Purpose

Pre-program PE studies are intended to thoroughly verify laboratory performance with sufficient time to address any problems that may be identified before initiating field sample collection. Pre-program PE studies are performed in advance of sample collection activities in order to establish laboratory proficiency in the use of project specific analytical procedures applied to the various sample types. Any unsatisfactory results reported by a given laboratory will be used proactively to identify the source of the deficiency and to institute appropriate corrective actions prior to collection and analysis of any NBSA field samples.

3. Description of Pre-Program PE Studies

All PE samples prepared will be categorized as “blind.” A “blind” PE sample is one where all necessary sample containers, labels, and COC will be completed based on the specifications for the field program, and sent as such to the laboratory for analysis. The source and certified analyte concentrations of the PE samples are not shared with the laboratory prior to reporting of sample results.

All standard reference materials (SRMs) used will be a certified reference material (CRM). This means the sample has been “characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainly, and a statement of metrological traceability.”[2]

Two types of PE samples will be utilized in this pre-program PE study, sediment/solid and tissue. The sediment/solid samples may either be a natural sediment/soil matrix material distinctly characterized to determine chemical constituents at measured concentrations, or a natural sediment/soil matrix fortified to provide specific analytical profiles. Tissue matrix PE samples are comprised of various species of fish, muscles and lobster hepatopancreas. Each of these tissue PE samples has been characterized via multiple analyses to establish specific analytical profiles and acceptance criteria.

Planned analyses, sources of SRMs, and laboratory participants are outlined in Table 1. Note that PE sample analyses include selected representative analytical procedures planned for use in the NBSA program. The scope of this study is based upon similar PE studies associated with recent investigations in the Lower Passaic River Study Area.

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[1] The pre-program performance evaluation study described here does not include performance evaluation studies that may be necessary in association with planned pore water sampling.

[2] http//www.NIST.gov/SRM/definitions.cfm

Table 1

Sediment Matrix Pre-Program Performance Evaluation Sample Summary			
Analytical Group	**Analytical Method**	**Vendor Name / Catalog Number**	**Laboratory**
PCDDs/PCDFs	USEPA Method 1613B	NIST/1944	eurofins/Lancaster Laboratories
PCB Congeners	USEPA Method 1668B	NIST/1944	eurofins/Lancaster Laboratories
Organochlorine Pesticides	USEPA Method 1699	phenova/Lot # 7051-14	Vista Analytical Laboratories
Semivolatile Organics (SIM)	USEPA Method Modified SW-846 8270	NIST/1944	eurofins/Lancaster Laboratories
Mercury	USEPA Method 1631	NRC/MESS-3	eurofins/Frontier Global Sciences
Methylmercury	USEPA Method 1630	IRMM/CC-580	eurofins/Frontier Global Sciences
Metals	USEPA Method SW-846 6020	ERA/540	eurofins/Lancaster Laboratories

Tissue Matrix Pre-Program Performance Evaluation Sample Summary			
Analytical Group	**Analytical Method**	**Vendor Name / Catalog Number**	**Laboratory**
PCDDs/PCDFs	USEPA Method 1613B	NRC/CARP-2	eurofins/Lancaster Laboratories
PCB Congeners	USEPA Method 1668B	NRC/CARP-2	eurofins/Lancaster Laboratories
Organochlorine Pesticides	USEPA Method 1699	NIST/1974c	Vista Analytical Laboratories
Semivolatile Organics (SIM)	USEPA Method Modified SW-846 8270	NIST/1974c	eurofins/Lancaster Laboratories
Mercury	USEPA Method 1631	NRC/DORM-4	eurofins/Frontier Global Sciences
Methylmercury	USEPA Method 1630	NRC/DORM-4	eurofins/Frontier Global Sciences
Metals	USEPA Method SW-846 6020	NRC/DORM-4 and TORT-3	eurofins/Lancaster Laboratories
Percent Lipids	USEPA Method 1613B	NIST/1946	eurofins/Lancaster Laboratories

ERA – Environmental Research Associates
IRMM – Institute for Reference Materials and Measurements
NIST – National Institute of Standards and Technology
NRC – National Regional Council of Canada
PCB – polychlorinated biphenyl
PCDD – polychlorinated dibenzo-*p*-dioxin
PCDF – polychlorinated dibenzofuran
SIM – selective ion monitoring

Attachment B

	CSL	CS1	CS2	CS3	CS4	CS5
PCB1	0.5	2	5	25	100	300
PCB2	0.5	2	5	25	100	300
PCB3	0.5	2	5	25	100	300
PCB4	0.5	2	5	25	100	300
PCB10	0.5	2	5	25	100	300
PCB9	0.5	2	5	25	100	300
PCB7	0.5	2	5	25	100	300
PCB6	0.5	2	5	25	100	300
PCB5	0.5	2	5	25	100	300
PCB8	0.5	2	5	25	100	300
PCB19	0.5	2	5	25	100	300
PCB14	0.5	2	5	25	100	300
PCB18+30	1	4	10	50	200	600
PCB11	0.5	2	5	25	100	300
PCB17	0.5	2	5	25	100	300
PCB12+13	1	4	10	50	200	600
PCB27	0.5	2	5	25	100	300
PCB24	0.5	2	5	25	100	300
PCB16	0.5	2	5	25	100	300
PCB15	0.5	2	5	25	100	300
PCB54	1	4	10	50	200	600
PCB32	0.5	2	5	25	100	300
PCB34	0.5	2	5	25	100	300
PCB23	0.5	2	5	25	100	300
PCB26+29	1	4	10	50	200	600
PCB25	0.5	2	5	25	100	300
PCB50+53	2	8	20	100	400	1200
PCB31	0.5	2	5	25	100	300
PCB20+28	1	4	10	50	200	600
PCB45	1	4	10	50	200	600
PCB51	1	4	10	50	200	600
PCB21+33	1	4	10	50	200	600
PCB46	1	4	10	50	200	600
PCB22	0.5	2	5	25	100	300
PCB52	1	4	10	50	200	600
PCB73	1	4	10	50	200	600
PCB43	1	4	10	50	200	600
PCB36	0.5	2	5	25	100	300
PCB49+69	2	8	20	100	400	1200
PCB39	0.5	2	5	25	100	300
PCB48	1	4	10	50	200	600
PCB104	1	4	10	50	200	600
PCB44+47+65	3	12	30	150	600	1800
PCB38	0.5	2	5	25	100	300
PCB59+62+75	3	12	30	150	600	1800
PCB96	1	4	10	50	200	600
PCB42	1	4	10	50	200	600
PCB35	0.5	2	5	25	100	300
PCB41	1	4	10	50	200	600
PCB40+71	2	8	20	100	400	1200
PCB37	0.5	2	5	25	100	300
PCB64	1	4	10	50	200	600
PCB72	1	4	10	50	200	600
PCB103	1	4	10	50	200	600
PCB68	1	4	10	50	200	600
PCB94	1	4	10	50	200	600

	CSL	CS1	CS2	CS3	CS4	CS5
PCB57	1	4	10	50	200	600
PCB95	1	4	10	50	200	600
PCB93+100	2	8	20	100	400	1200
PCB58	1	4	10	50	200	600
PCB98+102	2	8	20	100	400	1200
PCB67	1	4	10	50	200	600
PCB88	1	4	10	50	200	600
PCB63	1	4	10	50	200	600
PCB91	1	4	10	50	200	600
PCB61+70+74+76	4	16	40	200	800	2400
PCB84	1	4	10	50	200	600
PCB66	1	4	10	50	200	600
PCB55	1	4	10	50	200	600
PCB89	1	4	10	50	200	600
PCB56	1	4	10	50	200	600
PCB121	1	4	10	50	200	600
PCB60	1	4	10	50	200	600
PCB92	1	4	10	50	200	600
PCB80	1	4	10	50	200	600
PCB155	1	4	10	50	200	600
PCB152	1	4	10	50	200	600
PCB90+101+113	3	12	30	150	600	1800
PCB150	1	4	10	50	200	600
PCB83	1	4	10	50	200	600
PCB136	1	4	10	50	200	600
PCB99	1	4	10	50	200	600
PCB112	1	4	10	50	200	600
PCB145	1	4	10	50	200	600
PCB86+87+97+109+119+125	6	24	60	300	1200	3600
PCB79	1	4	10	50	200	600
PCB78	1	4	10	50	200	600
PCB85+116+117	3	12	30	150	600	1800
PCB110+115	2	8	20	100	400	1200
PCB81	1	4	10	50	200	600
PCB82	1	4	10	50	200	600
PCB148	1	4	10	50	200	600
PCB77	1	4	10	50	200	600
PCB111	1	4	10	50	200	600
PCB135+151	2	8	20	100	400	1200
PCB120	1	4	10	50	200	600
PCB154	1	4	10	50	200	600
PCB144	1	4	10	50	200	600
PCB147+149	2	8	20	100	400	1200
PCB134	1	4	10	50	200	600
PCB143	1	4	10	50	200	600
PCB108+124	2	8	20	100	400	1200
PCB139+140	2	8	20	100	400	1200
PCB107	1	4	10	50	200	600
PCB131	1	4	10	50	200	600
PCB123	1	4	10	50	200	600
PCB106	1	4	10	50	200	600
PCB142	1	4	10	50	200	600
PCB118	1	4	10	50	200	600
PCB132	1	4	10	50	200	600
PCB122	1	4	10	50	200	600
PCB188	1	4	10	50	200	600
PCB114	1	4	10	50	200	600
PCB133	1	4	10	50	200	600

	CSL	CS1	CS2	CS3	CS4	CS5
PCB179	1	4	10	50	200	600
PCB165	1	4	10	50	200	600
PCB105	1	4	10	50	200	600
PCB146	1	4	10	50	200	600
PCB184	1	4	10	50	200	600
PCB161	1	4	10	50	200	600
PCB176	1	4	10	50	200	600
PCB153+168	2	8	20	100	400	1200
PCB141	1	4	10	50	200	600
PCB186	1	4	10	50	200	600
PCB130	1	4	10	50	200	600
PCB127	1	4	10	50	200	600
PCB137	1	4	10	50	200	600
PCB164	1	4	10	50	200	600
PCB129+138+163	3	12	30	150	600	1800
PCB160	1	4	10	50	200	600
PCB158	1	4	10	50	200	600
PCB178	1	4	10	50	200	600
PCB175	1	4	10	50	200	600
PCB126	1	4	10	50	200	600
PCB128+166	2	8	20	100	400	1200
PCB187	1	4	10	50	200	600
PCB182	1	4	10	50	200	600
PCB183+185	2	8	20	100	400	1200
PCB174	1	4	10	50	200	600
PCB159	1	4	10	50	200	600
PCB162	1	4	10	50	200	600
PCB177	1	4	10	50	200	600
PCB202	1.5	6	15	75	300	900
PCB167	1	4	10	50	200	600
PCB181	1	4	10	50	200	600
PCB171+173	2	8	20	100	400	1200
PCB201	1.5	6	15	75	300	900
PCB156+157	1	4	10	50	200	600
PCB204	1.5	6	15	75	300	900
PCB197+200	3	12	30	150	600	1800
PCB172	1	4	10	50	200	600
PCB192	1	4	10	50	200	600
PCB180+193	2	8	20	100	400	1200
PCB191	1	4	10	50	200	600
PCB170	1	4	10	50	200	600
PCB190	1	4	10	50	200	600
PCB169	1	4	10	50	200	600
PCB198+199	3	12	30	150	600	1800
PCB196	1.5	6	15	75	300	900
PCB203	1.5	6	15	75	300	900
PCB208	1.5	6	15	75	300	900
PCB195	1.5	6	15	75	300	900
PCB189	1	4	10	50	200	600
PCB207	1.5	6	15	75	300	900
PCB194	1.5	6	15	75	300	900
PCB205	1.5	6	15	75	300	900
PCB206	1.5	6	15	75	300	900
PCB209	1.5	6	15	75	300	900
13C12-PCB28	100	100	100	100	100	100
13C12-PCB111	100	100	100	100	100	100
13C12-PCB178	100	100	100	100	100	100
13C12-PCB9	100	100	100	100	100	100

	CSL	CS1	CS2	CS3	CS4	CS5
13C12-PCB52	100	100	100	100	100	100
13C12-PCB101	100	100	100	100	100	100
13C12-PCB138	100	100	100	100	100	100
13C12-PCB194	100	100	100	100	100	100
13C12-PCB1	100	100	100	100	100	100
13C12-PCB3	100	100	100	100	100	100
13C12-PCB4	100	100	100	100	100	100
13C12-PCB8	100	100	100	100	100	100
13C12-PCB19	100	100	100	100	100	100
13C12-PCB15	100	100	100	100	100	100
13C12-PCB54	100	100	100	100	100	100
13C12-PCB32	100	100	100	100	100	100
13C12-PCB31	100	100	100	100	100	100
13C12-PCB104	100	100	100	100	100	100
13C12-PCB47	100	100	100	100	100	100
13C12-PCB37	100	100	100	100	100	100
13C12-PCB95	100	100	100	100	100	100
13C12-PCB70	100	100	100	100	100	100
13C12-PCB60	100	100	100	100	100	100
13C12-PCB155	100	100	100	100	100	100
13C12-PCB85	100	100	100	100	100	100
13C12-PCB81	100	100	100	100	100	100
13C12-PCB77	100	100	100	100	100	100
13C12-PCB123	100	100	100	100	100	100
13C12-PCB118	100	100	100	100	100	100
13C12-PCB188	100	100	100	100	100	100
13C12-PCB114	100	100	100	100	100	100
13C12-PCB133	100	100	100	100	100	100
13C12-PCB105	100	100	100	100	100	100
13C12-PCB141	100	100	100	100	100	100
13C12-PCB127	100	100	100	100	100	100
13C12-PCB126	100	100	100	100	100	100
13C12-PCB128	100	100	100	100	100	100
13C12-PCB162	100	100	100	100	100	100
13C12-PCB202	100	100	100	100	100	100
13C12-PCB167	100	100	100	100	100	100
13C12-PCB156+157	200	200	200	200	200	200
13C12-PCB180	100	100	100	100	100	100
13C12-PCB169	100	100	100	100	100	100
13C12-PCB208	100	100	100	100	100	100
13C12-PCB189	100	100	100	100	100	100
13C12-PCB205	100	100	100	100	100	100
13C12-PCB206	100	100	100	100	100	100
13C12-PCB209	100	100	100	100	100	100